

# **The Use of Tritium Autoradiography to Assess Fatigue Damage in Metals**

**K.A. Tsoi and A.K. Wong**

**DSTO-TR-1273**

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# **The Use of Tritium Autoradiography to Assess Fatigue Damage in Metals**

**K.A. Tsoi and A.K. Wong**

**Airframes And Engines Division  
Aeronautical and Maritime Research Laboratory**

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## **ABSTRACT**

This report introduces a technique known as tritium autoradiography which may be useful in the detection of fatigue damage in metallic components. Tritium autoradiography involves the infusion of radioactive hydrogen (tritium) into a fatigue damaged structure. The damaged areas of the structure may act as preferred trapping sites for the tritium and as the tritium decays via the emission of beta particles it can be detected using nuclear emulsion film.

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## The Use of Tritium Autoradiography to Assess Fatigue Damage in Metals

### EXECUTIVE SUMMARY

During the 1950s a series of aircraft accidents involving the Comet aircraft made the aircraft industry much more aware of the problems associated with the fatigue damage of metals. Although a lot more is now known about fatigue, there still exists a great need to know where and when a structure will fail. Over the years a number of different techniques have been developed for the purpose of detecting fatigue-induced damage in order to determine where and when an aircraft will fail structurally. This report describes a technique known as tritium autoradiography which involves the infusion of radioactive hydrogen (tritium) into a structure that may contain fatigue damage. The damaged parts of the structure are likely to act as a preferred trapping site for the tritium and as the tritium decays via the emission of beta particles it can be detected using nuclear emulsion film. This report describes the technique in detail. Preliminary results indicate that this technique does show some degree of promise for the early detection of fatigue damage. Initial autoradiographs of fatigued aluminium 2024 specimens show that some sort of trapping of the tritium is occurring and a distinct difference is observed between the autoradiographs of the fatigued and unfatigued specimens. Further work is required to obtain more accurate autoradiographs, to determine, more precisely, what is causing the trapping and which areas, shown up as darker regions in the autoradiograph, are due to fatigue damage in the structure.



## Authors

**Kelly A. Tsoi**  
*Airframes and Engines Division*

Kelly Tsoi completed a Bachelor of Science Honours degree majoring in physics at the School of Physics at The University of Melbourne in 1995. She commenced work at AMRL in January 1996 and has worked on methods of fatigue life enhancement using smart materials. She is currently a Cadet Research Scientist investigating the behaviour and applications of shape memory alloy composites.

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**Albert K. Wong**  
*Airframes and Engines Division*

Albert Wong holds a Bachelor of Engineering (Hon 1) and a PhD in Mechanical Engineering from the University of New South Wales. Since joining DSTO in 1985, he has worked in a number of research areas that include: fracture mechanics, finite element methods, and experimental stress analysis. In particular, he has made major contributions in the fundamental understanding of thermoelastic stress analysis, and is the co-developer of the world's first focal-plane array stress analysis system based on an infrared imager. He is currently a Principal Research Scientist in AMRL's Airframes and Engines Division, and heads the Machine Dynamics area that is primarily concerned with research into Health Monitoring of rotating machineries using vibration analysis techniques.

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## 1 Introduction

During the 1950s a series of aircraft accidents involving the Comet aircraft made the aircraft industry much more aware of the problems associated with the fatigue damage of metals. Although a lot more is now known about fatigue, there still exists a great need to improve on our ability to predict where and when a structure will fail. Over the years a number of different techniques have been developed for this purpose. Much work has been done on finding new and improved techniques with which to detect fatigue cracks earlier and more reliably and, in particular, to detect the damage without compromising the structure in any way. Such techniques are generally known as Non Destructive Inspection (NDI) techniques.

To date the minimum crack size that can typically be detected reliably using conventional NDI tools is approximately 0.8 mm, as can be seen from Figure 1. This size can be achieved using magnetic particles although this requires both ideal conditions and the best equipment, both of which aren't always available.

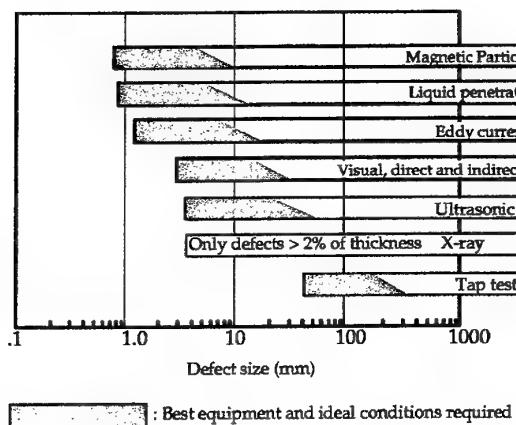


Figure 1: From ESDU data, (1)

A report entitled "Methods of Early Fatigue Damage Detection" [1] discusses existing NDI techniques as well as several new techniques which are being investigated, including the mean stress gauge and tritium autoradiography. Tritium<sup>1</sup> autoradiography can, in principle at least, provide a much earlier warning on potential failure sites and the degree of fatigue damage compared to conventional NDI techniques. It can potentially highlight flaws that may not be observable using techniques such as the magnetic particle technique, eddy current, liquid penetrant or X-ray radiography.

Autoradiography was first discovered in 1867 by Niepce de St Victor [2] with the use of silver chloride and iodide emulsions to investigate uranium nitrate and tartrate. The emulsions blackened with exposure to these uranium salts. Twenty nine years later, radioactivity was discovered by Becquerel. This major discovery was made using a technique

<sup>1</sup>Tritium is a radioactive isotope of hydrogen.

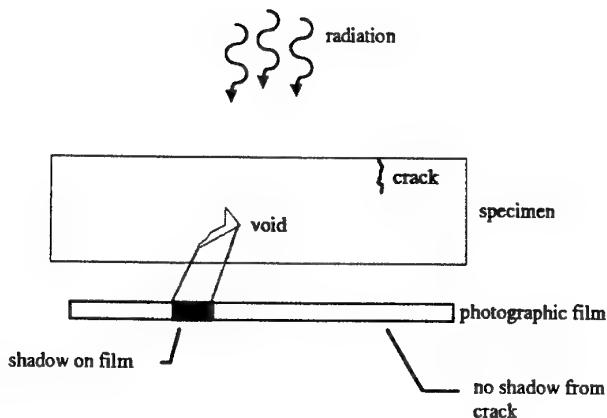


Figure 2: Radiography. (After ESDU 1991)

that was later to be known as autoradiography. It wasn't until 1924 that autoradiography was used as a scientific technique, by Lacassagne [2], to study polonium distribution in biological specimens. Today autoradiography is used to study biological and other processes. In material science in particular, it is used for the study of hydrogen embrittlement of metals. The use of tritium autoradiography to study defects in metals was first looked at in 1959 to aid in the understanding of hydrogen embrittlement in metals. Tritium was used because it has essentially the same properties as hydrogen, but its radioactivity provides a convenient means for marking its extent and location of entrapment within the metal.

The purpose of this report is to present the results of an investigation into the use of tritium autoradiography as an early detector of fatigue induced damage. It will briefly discuss the methods of autoradiography and the techniques used in this and previous investigations. The results of this investigation will be presented and discussed.

## 2 Autoradiography

### 2.1 Radiography

Radiography is frequently used in both medical and mechanical fields as a means of visualising the internal structure of an object. In general, it involves first irradiating the object in question by an extraneous source of high energy radiation. This radiation travels through the object, and is differentially absorbed depending on the thickness, composition, density and defect structure of the object [3]. A photographic film placed on the opposing side of the object is exposed to the transmitted radiation and an image of the internal structure, including defects, is obtained. This is known as film radiography, and the process is depicted in Figure 2.

The most common radiation types used in radiography are X-ray and gamma ray radiation, though neutron radiography is also in use. The major distinction between the three

types is the penetration depth, with X-rays being the least penetrative and neutrons the most.

As an NDI tool, current radiographical techniques are acknowledged as having a relatively poor defect sensitivity. For example, it is stated in [4], that X-ray radiography is only capable of reliably detecting defects greater than about 3 mm in size, and only those defects which are greater than 2% of the thickness of the specimen. Furthermore, it is noted that surface cracks need an opening of at least 0.03 mm to be detected, meaning that closed cracks cannot be detected.

Apart from using an extraneous source of radiation, it is possible to visualise defects by introducing radioactive isotopes into the structure itself. During the investigation into the Comet aircraft accidents [5], it was found, quite by accident, that radioactive isotopes introduced into the fuel, as a means of detecting fuel leakage, accumulated in a minute crack in the combustion chamber. Consequently, a crack that was too small to be detected by eye was discovered. This example highlights the possibility of using radioactive isotopes as detectors of fatigue damage. Such a possibility could be realised if combined with autoradiographic techniques.

## 2.2 Radioactivity and Beta Decay

As was mentioned in the introduction, radioactivity was first discovered in 1896 by Becquerel, who concluded from observing a darkening of photographic plates after exposure to uranium crystals, that a new type of radiation was being spontaneously produced by the uranium.

Radioactivity is the spontaneous decay of the nuclei of a substance via the emission of an alpha particle ( ${}^4\text{He}$  nucleus), a beta particle (either electrons or positrons), or a gamma ray (high energy photons). The decay of nuclei via any of these processes can be described by the radioactive decay law:

$$N(t) = N_0 e^{-\lambda t} \quad (1)$$

where

$$\lambda = -\frac{dN}{dt} \frac{1}{N} \quad (2)$$

and is known as the disintegration or decay constant and  $N$  is the number of nuclei.

The half-life of a substance is the time required for half of the nuclei to decay and is given by

$$t_{\frac{1}{2}} = \frac{\ln(2)}{\lambda} \quad (3)$$

The activity of a radioactive sample is measured using the curie (Ci) and the becquerel (Bq), where  $1 \text{ Ci} = 3.7 \times 10^{10} \text{ decays/sec} = 3.7 \times 10^{10} \text{ Bq}$ .

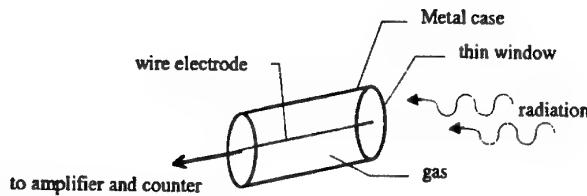


Figure 3: Diagram of a Geiger counter.

Tritium undergoes beta decay, whereby electrons ( $\beta^-$ ) are emitted from the radioactive substance (ie., the tritium nucleus). This comes about when there is enough energy from the decay process to create an electron within the nucleus of the atom. The electron is then emitted from the nucleus. The decay processes can be described as:  
 $n \rightarrow p + \beta^- + \bar{\nu}$ , negative beta decay ( $\beta^-$ ) where n is a neutron, p is a proton and  $\bar{\nu}$  is an antineutrino (the anti particle of a neutrino).

Beta particles can be detected using a variety of methods. The most common form of radiation detection is the Geiger Müller tube. The simplest Geiger counter consists of a cylinder containing a low pressure gas and two electrodes, as shown in Figure 3. Incoming radiation ionizes the gas that produces a current flow between the electrodes.

The sensitivity for such devices is, however, inadequate for the present work. As was seen in the work by Saitoh *et al* [6], the size of dislocations within the metal are of the order of microns. To obtain any useful information about the distribution of tritium concentrations, the detectors must have extremely accurate resolution. Another problem which arises is the fact that beta radiation from the decay of tritium only travels 4.5 - 6 mm in air [7] or through 0.01 mm of paper [8]. By the time a beta particle travels from a void within the metal, through the metal window of the counter to the low pressure gas, it is likely that the beta particle will have lost all of its energy and hence cannot be detected.

Liquid scintillators are also used in the detection of beta particles. The principle behind liquid scintillation is that a specimen containing radioactive substances is placed into a scintillation cocktail, which contains a fluor. The radioactive substances decay in this cocktail and the energy of the decaying particles is converted into light by way of excitation of the fluor molecule, which emits visible light when returning to its ground state. This light then travels down a photomultiplier tube and is converted into an electrical pulse, the height of which corresponds to the energy of the original ionising particle. The problem with liquid scintillation is that only an overall measurement of a radioactive substance is obtained for a sample. For this study high spatial resolution is required such that the location of the tritium particles can be determined within the specimen, and so renders the liquid scintillation method inappropriate. The ideal system to use in this case is autoradiography.

## 2.3 Tritium Autoradiography

Autoradiography is a micrograph technique by which radiation emanating from a body may be detected [9]. Figure 4 describes this process.

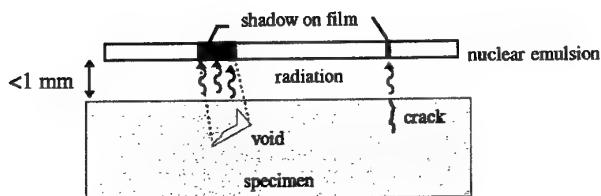
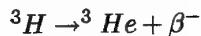


Figure 4: Autoradiography.

The main idea is that the specimen is infused with a radioactive isotope and the decay of the isotope is then detected using photographic emulsions. Autoradiography is currently used as a technique to study biological processes as well as to study hydrogen embrittlement in metals. This technique has the advantage that the spatial distribution of the hydrogen can be recorded with great accuracy.

The isotope most often used in these investigations of hydrogen embrittlement in metals is tritium. Tritium is a radioactive isotope of hydrogen and decays to helium by emitting a negative beta particle (ie., an electron). The decay process is:



Tritium has similar properties to hydrogen, and therefore diffuses and becomes trapped in the same regions in the microstructure as hydrogen does. It can thus be used to trace the concentration and distribution of hydrogen within metals.

Tritium emits beta particles that have energies ranging from 3.6 keV to a maximum energy of 18.61 keV, which are relatively low and, consequently, the particles have poor penetration ability. In fact these particles are stopped by as little as 4.5 mm of air [7] and 0.01 mm of paper [8]. As a result, beta radiation is difficult to detect and a photographic detection system must be used on or very close to the surface of the specimen.

There are several possible autoradiographic techniques that can be used, two of which are described below. The first is known as contact autoradiography. The specimen containing the tritium is placed in direct contact with a dry photographic plate. This is a macro-technique and is useful for obtaining information about the distribution and concentration of tritium within the specimen. After exposure, the plate is removed from the specimen and developed. The second technique is known as electron micro-autoradiography and involves a liquid emulsion. The photographic emulsion is developed whilst still in contact with the specimen and the resulting autoradiograph is examined using either a scanning or transmission electron microscope. This technique is suitable for studying tritium distributions on a microscopic scale, for example, looking at tritium distribution between phases and boundaries, or looking at the area of high dislocation concentration just ahead of a crack tip. An explanation of other techniques is described by Caskey, [7].

Aucouturier *et al* [10], used the technique of charging tritium into both maraging steel and FeTi alloy specimens. The specimens were then polished down to thin foils and

coated with a collodion solution to prevent oxidation. A nuclear photographic emulsion was then applied to the surface of these specimens and the emulsion was then exposed for ten days at -30°C. This differed from the previously more conventional approach in that a carbon replica of the surface of the specimen was made, the emulsion placed onto this surface and an electron-micro-autoradiograph then taken. Aucouterier *et al* found that the FeTi alloy systems show a variety of trapping processes including trapping on carbide matrix interfaces, grain boundaries and dislocation boundaries. It was also found for maraging steels that trapping occurred on inclusion interfaces, grain boundaries and plate boundaries.

Asaoka *et al* [11], also did a study of the cracking behaviour observed after cathodically charging tritium into a specimen of an FeTi alloy (0.15 Wt% Ti). Four types of traps were observed. 1. precipitate matrix interfaces, 2. grain boundaries, 3. dislocation segregation and 4. plate like and needle like precipitate-matrix interfaces. It was also found that intergranular cracks, caused by a high charging current density (10mA/cm<sup>2</sup> over 10 hours), trapped the tritium atoms which were easily observed using tritium autoradiography. It was also found that specimens that were charged with a low current density had formed no cracks.

It was determined by Saitoh *et al* [6], that the spatial resolution for autoradiography of beta particles travelling through a photographic emulsion and aluminium was better than 1 mm. Saitoh *et al*, [6], charged tritium into specimens of pure aluminium, Al-1 mass% Mg<sub>2</sub>Si alloy and Al-4 mass % Cu alloy, using a cathode charging method. A sensitive photographic film, containing a layer of fine AgBr grains, was adhered to a thin collodion film and then placed onto the specimen and exposed to the beta rays emitted from the tritium in the sample. The film was developed and the resulting micro-autoradiograph was examined, *in situ*, with the specimen, using a transmission electron microscope (TEM). The samples used in these experiments were 0.2 mm thick and ranged from 4 to 10 mm in diameter. The results of these experiments showed that for the pure aluminium and Al-1 mass% Mg<sub>2</sub>Si alloy, the dislocations act as traps for the hydrogen as well as a short circuiting diffusion path. Similar results were also obtained by Iijima *et al* [12].

When compared to other defect detection systems, tritium autoradiography has a resolution that is far superior [6]. Up until 1991, the best resolution for detection of defects was found using magnetic particle techniques. A surface breaking or shallow sub surface defect of 0.8 mm can be detected in this way. There have been many methods used in the study of hydrogen embrittlement that have involved cathodically charging hydrogen into a specimen and then either observing the degree of hydrogen entrapment, or straining the material and testing the level of embrittlement of the material.

The idea behind the present investigation is to determine whether there is any correlation between tritium concentration and fatigue life. As has been observed previously, tritium autoradiography is capable of imaging defects such as grain boundaries within a metal, with a resolution of greater than 1 mm. Generally, when a metal is fatigued, voids and dislocations grow in areas of high stress concentration. In these areas it is more likely that cracks will develop. As was mentioned previously, it is well known that hydrogen becomes trapped in these voids and dislocations and tritium has also been shown to be attracted to the grain boundaries in metals. Thus, it may be possible to use this in order to detect areas of high fatigue damage and cracking. By taking autoradiographs of a series

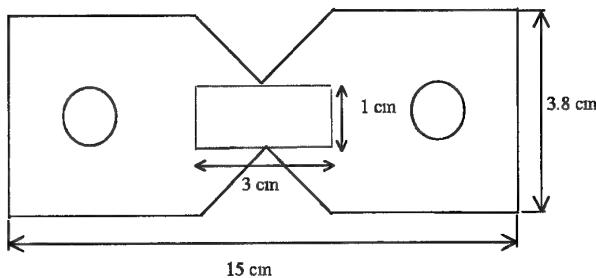


Figure 5: *Butterfly-like specimen.*

of specimens that have been fatigued at various loads, the distribution of tritium within the specimens can be determined and correlated with the fatigue damage/cracking within a specimen.

### 3 Experimental Techniques

#### 3.1 Specimen Preparation

The specimens were made from the aluminium alloy 2024, and pure aluminium. These materials were used due to their application within the aerospace industry. Pure aluminium was used because it contains few impurities. This would initially, allow us to observe tritium distribution without having to worry about the tritium becoming trapped by any impurities. The specimens were 1 mm thick and were cut into a butterfly-like specimen as shown in Figure 5. This shape was chosen so that a variable stress range could be obtained over a small distance and, hence, the distribution of tritium within the specimen would be much more defined, with a greater amount of tritium expected in regions of high stress concentration.

One side of the specimens was then polished to a 1 mm finish by either using progressively finer abrasive paper, and finally with 1 mm diamond paste or by means of a micro milling machine. The 2024 aluminium alloy specimens had a thin cladding of pure aluminium that was removed before polishing, so that a difference between the specimens could be observed. These specimens were then fatigued using a 50 kN servohydraulic testing machine with an amplitude of 7 kN, a DC offset of 8 kN and a peak to peak load of 14 kN. This was sufficient to produce the significant degree of fatigue damage, with some specimens producing tiny hairline cracks at the notch-corners of the specimen, which were visible to the eye. By using these specimens, there is a greater knowledge as to where the tritium should become trapped.

The specimens were then cut out along the dotted line, as shown in Figure 5, using a diamond saw. Care was taken to ensure that the surface of the specimens was not scratched and photographs of the surface were taken before and after cyclic loading as well as after the cutting process.

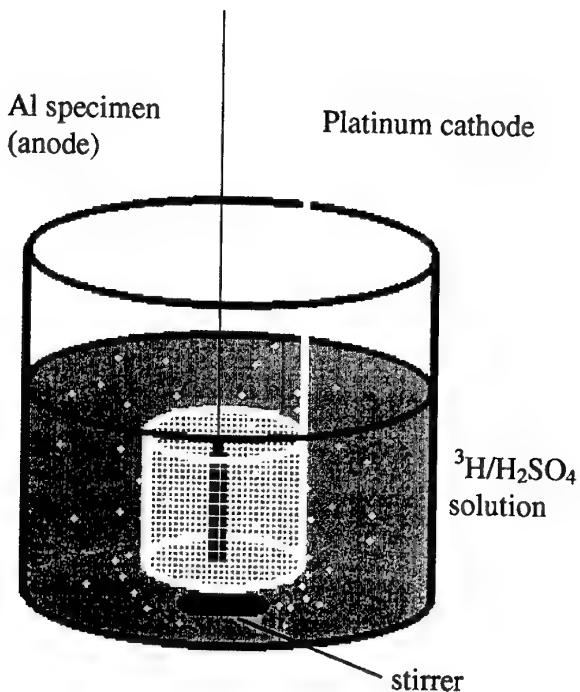


Figure 6: Experimental setup for charging.

## 3.2 Specimen Charging

### 3.2.1 Tritium Solution Preparation

Due to the hazardous nature of tritium and tritiated water, in particular, a safety study of how to handle the tritiated water was completed and can be found in Appendices A, B and C. A solution containing 0.5 N sulfuric acid ( $H_2SO_4$ ) and tritiated water was prepared such that the solution had an activity of 10-3 Ci/ml. The 1000 ml of the original tritiated water solution had an activity of 0.5 Ci. 13.33 ml of concentrated  $H_2SO_4$  was then mixed with it and 200 ml of the resulting solution was then used during the charging process.

### 3.2.2 Charging Process

Before the charging of the specimen commenced, the back and sides of the specimen were coated with lacomit, in order to prevent tritium penetration through surfaces other than the front (polished side) of the specimen. Once 200 ml of the solution was placed into a beaker, the specimen was attached to an alligator clip and attached to a DC power supply and a platinum cathode was used such that the area of the cathode was much greater than that of the area of the specimen to be charged. This avoids overheating of the specimen. The experimental setup can be seen in Figure 6.

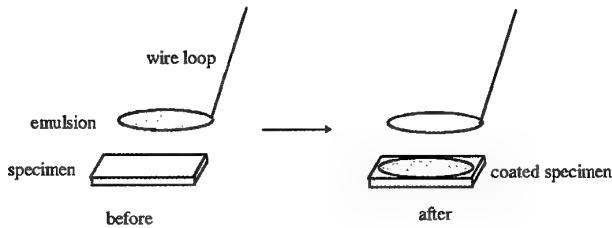


Figure 7: Wire loop method.

The surface area of the specimen was measured and the current to be used was calculated using Equation 4,

$$I = SA \times \rho \quad (4)$$

where  $SA$  is the surface area of the specimen, measured in  $\text{m}^2$  and  $\rho$  is the current density in  $\text{A}/\text{m}^2$ . A current density of  $100 \text{ A}/\text{m}^2$  was used. Charging of the specimen occurred for 1 hour. The specimen was then placed into a light proof black box, and into a freezer at  $-10^\circ\text{C}$  for 1 day, to allow any surface hydrogen/tritium to diffuse off.

### 3.3 The Emulsion Process

The emulsions used in this investigation were Ilford K2 and L4 emulsions. The K2 emulsion has a crystal diameter of 0.2 mm and is used for light microscopy and the L4 emulsion has a crystal diameter of 0.11 mm and is best for electron microscopy due to its higher resolution. The preparation for both emulsions is identical. Everything must be done under safelight conditions, where the safelight used was an Ilford 902 (light brown) for general darkroom illumination. The emulsion was placed into a beaker and heated in a water bath held at  $40^\circ\text{C}$ . The emulsion was allowed to melt undisturbed for 1 hour followed by gentle stirring so as not to create any air bubbles, as once air bubbles have been formed, the emulsion must be strained to remove them. The emulsion was diluted with distilled water (between 1-4 ml) until the desired consistency was reached and subsequently removed from the bath and allowed to semi-gel at room temperature. A Ni-Cr wire loop with a diameter sufficiently large to cover the area of interest on the specimen (see Figure 7) was dipped into the emulsion, creating a "stable" loop. This step determines whether the correct consistency has been reached. A "stable" loop is where the emulsion doesn't flow or is not uneven in thickness. It should, when held up to a safe light, have a consistent transparency.

The emulsion was then used in one of two methods. The first method used the idea of electron micro autoradiography, as was mentioned in Section 2.3, to apply the emulsion directly to the specimen. The specimen was mounted onto a holder, for ease of handling, using double sided tape. After the emulsion was applied to the specimen it was placed into a light proof black box and into a freezer at  $-10^\circ\text{C}$  for a specified length of time. The second method used the idea of contact autoradiography. The idea was to apply the emulsion to a clean glass slide. The glass slides were thoroughly washed in Decon 90 and

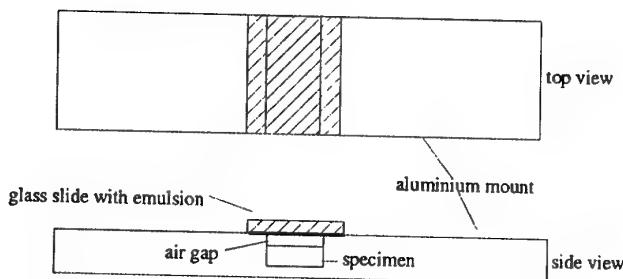


Figure 8: Specimen mount for contact autoradiography.

then rinsed in distilled water and dried off with lens tissues. The emulsion was placed onto the slides, using the wire loop method, and allowed to dry. These slides were stored in a light proof black box in the refrigerator. To use the slide, a specimen was attached to the aluminium mount using double sided tape as shown in Figure 8.

The glass slide with the emulsion was then placed over the specimen, emulsion side down and fastened to the specimen using double sided tape. The mount was then placed in a black, light proof box and placed into a freezer at -10°C for a specified amount of time. Both versions of autoradiography were attempted. However it was found that the electron micro autoradiography was far better. The contact autoradiography was attempted on several specimens however the glass slides would crack during attempts to remove them after exposure. Therefore the glass slide was developed while still in contact with the aluminium mount. However it is doubtful that the developer actually contacted the emulsion side of the glass slide since the air gap between the slide and the specimen was too small to allow the liquid in. The glass slides cracked completely during attempts to remove them from the mount (to observe the results). For the majority of the experiments electron micro autoradiography was used.

### 3.4 Development of the Emulsion

Once the emulsion has been exposed for a specified time the slide/specimen was removed from the light proof box. The emulsions were developed in Kodak D-19 developer for 4 minutes at 20°C. For the Ilford K2 emulsion, the developer was diluted with distilled water at a concentration of 1:1 whereas for the Ilford L4 emulsion, the developer was used undiluted. An acid stop bath of 1 part Ilford IN-1 to 39 parts distilled water was then used, and the slide/specimen with the emulsion was then fixed using one part Ilford Hypam mixed with four parts distilled water for 4 minutes at room temperature. The slide/specimen was then washed in water for 10 minutes at a temperature similar to that of the processing solutions. The developed slides/specimens were then stored in a dark box in the refrigerator. The specimens could then be examined using a Reichert-Jung MeF3 light with various degrees of magnification.

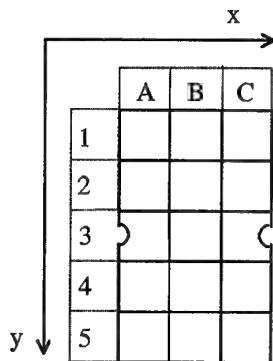


Figure 9: Specimen cutout.

### 3.5 Other Experiments Undertaken

#### 3.5.1 Examination of Tritium Content using a Liquid Scintillation Technique

Before specimens were charged with tritium and autoradiographs taken of them, a simple experiment was conducted to test how much tritium was in a charged specimen and how the tritium was roughly distributed throughout the specimen. A liquid scintillation counter was used to do this. The principle behind liquid scintillation counting was described in Section 2.2. Although liquid scintillation gives only bulk measurements of how much tritium is in a specimen, it was possible to obtain some idea of where the tritium was being distributed throughout the specimen by cutting the specimen into pieces and then looking at each piece separately.

A specimen was charged, as was described in Section 3.2.2. After charging was completed the specimen was cut and labelled, an example of which is shown in Figure 9.

The pieces were then placed into a vial containing 10 ml of the liquid scintillation cocktail, Readysafe. These were then held for 24 hours to allow the tritium gas to diffuse out of the specimen pieces and dissolve into the cocktail. The vials were then placed into the Beckman LS 5801 liquid scintillator counter and initially each vial was counted for 10 minutes. The number of counts per vial was then determined.

## 4 Results

### 4.1 Examination of Tritium Content

As mentioned in Section 3.5.1, before completing the autoradiographs, it was desirable to determine the amount of tritium that was charged into the specimens as well as to determine the distribution of the tritium throughout the specimen. The process described in Section 3.5.1 was carried out on an unfatigued and fatigued specimen, both of which had been charged in the tritium solution. The results for the unfatigued specimen, where

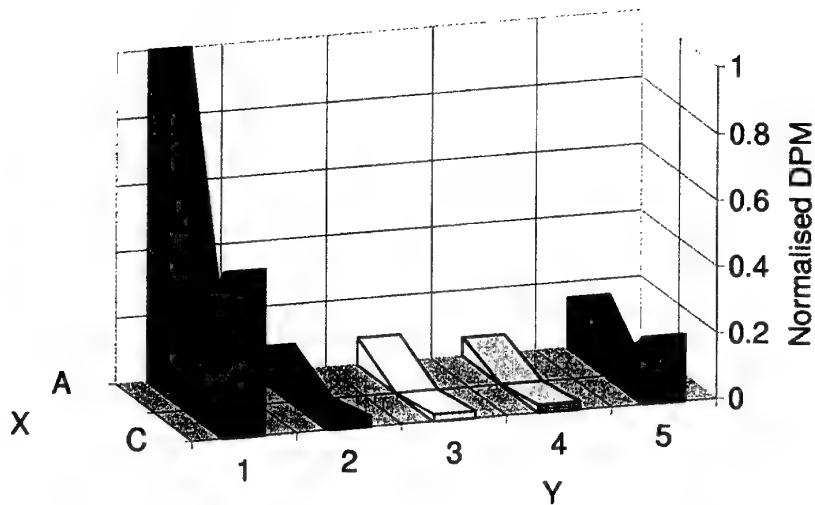


Figure 10: Liquid scintillation for unfatigued specimen.

the disintegration rate has been normalised by the maximum value, can be seen in Figure 10.

The x and y axes correspond to those shown in Figure 9. From this graph it can be seen that along the centre region of the specimen there were very little counts observed, the majority of the counts occurring along the far edge of the specimen. This would indicate that the tritium was somehow caught along that edge, perhaps due to an uneven coating of lacomit. It could also be caused by seepage between the lacomit/Al interface, thus causing a greater surface area around the edges and hence producing a greater concentration of tritium in these areas. The results for the fatigued specimen are shown in Figure 11. This chart shows a significantly different distribution compared to that for the unfatigued specimen and suggests a much higher concentration of tritium, particularly along the centre-line region. The very high values at the corners of the specimen may be due to the same problem of the lack of lacomit coverage as previously mentioned. Several other specimens which were fatigued and charged also followed similar trends.

## 4.2 Tritium Autoradiography

Figure 12 shows a micrograph of the resulting autoradiograph for a specimen that was charged and then a tritium autoradiograph taken, as described in Section 3. This specimen had a crack of approximately 0.5 mm long in the region indicated in Figure 12.

From this photograph it can be seen that within a semi-circular region surrounding the tip of the crack, there is a series of black spots suggesting that this could be where the tritium has become trapped. Comparing this photograph to that of an autoradiograph of an unfatigued and uncharged specimen, Figure 13, it can be seen that the dark spots in the

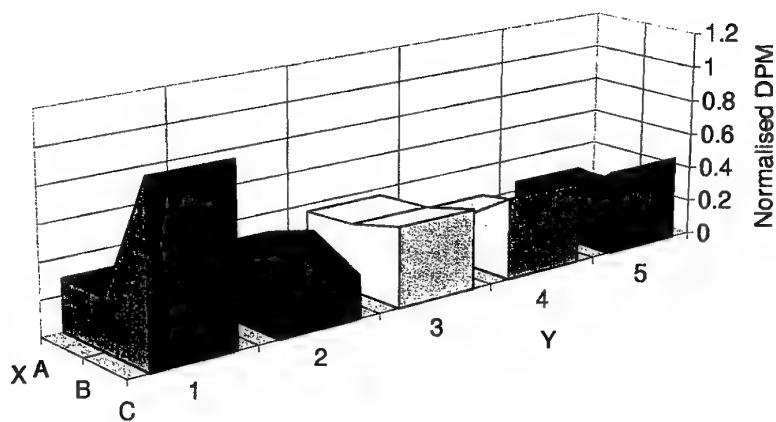


Figure 11: Liquid scintillation of fatigued specimen.

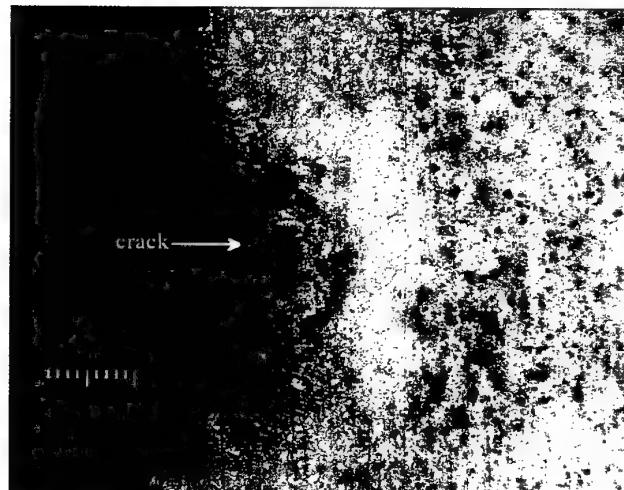
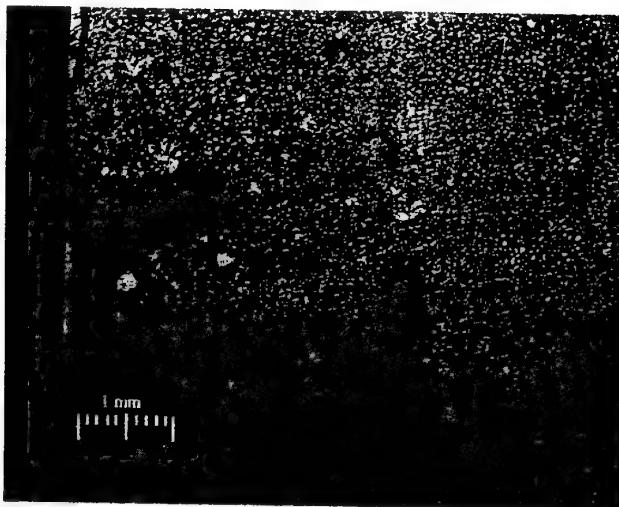


Figure 12: Tritium autoradiograph of Al 2024 butterfly specimen, fatigued and charged. (Autoradiographs such as this were taken of the notch area of pieces 3A or 3C as shown in Figure 9).



*Figure 13: Tritium autoradiograph of Al 2024 butterfly specimen, unfatigued and uncharged.*

previous figure aren't visible, due to a lack of tritium in the specimen. Another experiment was later completed whereby an unfatigued and charged specimen had an autoradiograph taken and the results can be seen in Figure 14.



*Figure 14: Tritium Autoradiograph of Al 2024 specimen, unfatigued and charged.*

Comparing this with the autoradiograph in Figure 12 shows that the experiment has been able to detect differences between the different specimens. At present it isn't clear what has caused the trapping of the tritium in these areas. However it is evident that there is a distinct difference between the fatigued and unfatigued specimens. Further work needs to be undertaken to establish a firm correlation between the amount of tritium entrapment and the level of fatigue damage over a broad range of loading conditions. To achieve this, however, a more efficient method is necessary, as the techniques employed in

this current work were both painstaking and time-consuming.

## 5 Conclusions

At the present time, it appears that the method of tritium autoradiography shows some degree of promise for the early detection of fatigue damage in metallic components. The results of the autoradiographs of charged and uncharged specimens has shown that there is a marked difference in the areas of tritium concentration, and that the tritium is infused into the regions of higher fatigue damage. Further work is required to pinpoint the exact regions of fatigue damage more accurately and to gain a better understanding of this method.

Possible further work includes the determination of a method to reduce the time it takes to obtain an autoradiograph. Perhaps by using different radioisotopes that emit stronger radiation but which also fit into vacancies and defects in metals without damaging the material.

It might also be possible to use a wetting agent on the surface of a specimen in order to absorb tritium into the surface instead of using the charging technique. This is expected to be more applicable to surfaces that have been fatigued to a stage where microcracks are already present.

## 6 Acknowledgments

The authors would like to thank David Rowlands for his invaluable contribution to the difficult experimental work that was carried out during this investigation. Thanks also goes to Noel Goldsmith for his help and advice using the optical and scanning electron microscopes, Dr Peter Grey for his help and advice on the use of the liquid scintillation counter, Alan Cox for his help in the micro milling of the specimens and Michelle Wakelam of Monash University for her help and advice on the handling and monitoring of tritium.

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## Appendix A: Safety Study Use of Radioactive Hydrogen (Tritium)

### A.1 Task 96/088 18 November 1996: General Considerations

Part of the work conducted under task 96/088 included the use of small amounts of radioactive hydrogen (tritium),  ${}^3\text{H}$ . This study details the measures that were adopted in order to minimise exposure of personnel and prevent accidents.

#### Tritium Hazards

Tritium ( ${}^3\text{H}$ ) is a low energy (18.6 keV) beta particle emitter with a half life of 12.3 years. Generally, the hazard from direct irradiation is small since the beta particles are stopped by 6 mm of air. The main hazard is via absorption through the skin or by ingestion or inhalation.

Since tritium is an isotope of hydrogen, it has the same chemical properties as hydrogen. Tritium readily undergoes isotopic exchange with hydrogen, especially in water and hydrocarbons, where a radioactive hydrogen atom is exchanged for a non radioactive hydrogen atom in the material which is in contact with the tritium. Hence, tritium can be readily absorbed by the skin. Tritium gas or tritium oxide may be absorbed through the lungs and skin to produce tritiated water in the body, thus presenting a hazard to all body organs. Taking into account its rate of excretion from the body, the effective half life of tritium in the body is about 10 days.

Tritium diffuses rapidly and it strongly adsorbs on to and absorbs into all surfaces, particularly construction materials. The adsorbed or absorbed tritium may be released later (out-gassing) and may represent a hazard long after the original tritium source is removed. Tritium is explosive at the appropriate concentrations. Because of the low energy of the beta particles, specialised radiation monitors are required to detect tritium.

#### Receipt, Storage And Disposal

##### Location of Use.

The experimental work involving the use of the tritium isotope was only conducted in Room 43 (now the smart structures laboratory) of Building 2 of the Fishermens Bend site. This laboratory was used by staff working on task 96/088. Keys to this laboratory were held by D. Rowlands, K. Tsui and T. Ryall. A keys register was established to keep track of those who were in possession of such keys and access during the experiments was strictly limited to those directly involved only.

The areas which the isotope were stored and used are defined in Figure A1.

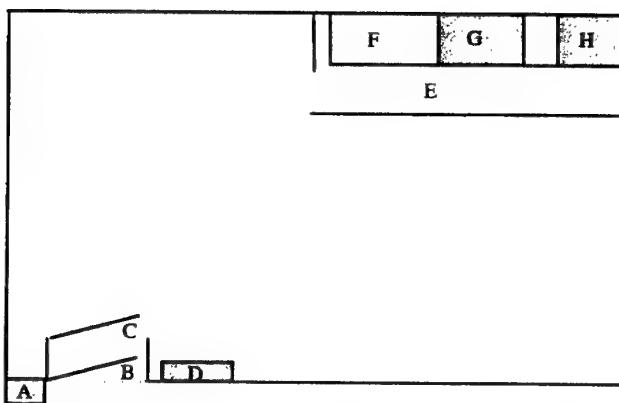


Figure A1: A. Flashing red safety light, B. Outer door, not locked, C. Inner locked door (opened by key only), D. Lockable refrigerator containing tritiated water, E. Dark room area where experiment will be undertaken, F. Workbench with taps and sink, G. Lockable fume cupboard with extraction fan to atmosphere and H. Lockable steel cabinet.

### Receipt

The radioactive isotopes were received from the supplier in a shielded container marked with the activity and volume. The isotopes were supplied in an ampoule of 0.2 ml in size. This container was stored in a locked refrigerator, Figure A1 (D), until required. The refrigerator containing the isotopes was labelled appropriately. The key to this refrigerator was held only by those working directly with the isotopes.

### Storage

The isotopes were diluted in distilled water and stored in 500 ml glass bottles. The bottles were labelled as radioactive, with a warning label and a description of the isotopes and locked in the refrigerator.

### Liquid Waste Disposal

The liquid waste was diluted with water until its maximum concentration fell below the legal limits for disposal down the sink (see Appendix C for the legal limits). The volume of water used for all rinsing and dilution was monitored either by measured amounts or via the use of a flow meter attached to the tap.

### Solid Waste Disposal

After the dilution process and the first of the series of experiments, the equipment and laboratory bench space was wipe-tested by Monash University researchers. The results were taken as a reference and a guide for future disposal of waste since this initial process presented the most severe conditions over the suite of experiments. If the results showed that the gloves and pipettes had a contamination below the legal limit (see Appendix B) they were disposed of using the normal disposal channels. If the results showed that the contamination limits of the experimental apparatus was above the legal disposal limits

they were stored in a locked metal cabinet (H) in a heavy duty plastic garbage bag and disposed of by a licensed waste disposal company. The ampoule in which the isotopes were supplied were stored together with the distilled tritium solution (see Section A.2) for the duration of the experiments. If the experiments were terminated before the 500 ml of solution was used up and it would take too long for it to be diluted and disposed of below the legal limits, a certified waste disposal company would have been used to dispose of the waste. If, by the end of the experiments, the liquid was used up, the amount of liquid left over would have been approximately 1.8 ml (based on experiments already undertaken to investigate the amount of liquid remaining within the bottle after being emptied). The empty jar together with the ampoule were then filled with 500 ml of water to obtain a radioactivity of approximately 1.8 mCi, which can be disposed of down the sink (see Appendix C). This rinse process should result in a residual activity of approx. 6.5 mCi within the jar. As a further precaution, a second rinse will be performed which should leave the jar (and ampoule) with a residual activity of approx. 0.02 mCi. This level is well below the specified limits and the apparatus can then be treated as ordinary waste. The volumetric flask which was used to dilute the radioactive isotope was diluted with water in a similar fashion until the radioactive level was well below the required limits and was disposed of similarly (see Appendix C).

### **Accounting**

The amounts of tritium used were accounted for in a laboratory log book, stating date, amount used, activity used, disposed waste, and any other information.

### **Contamination Monitoring**

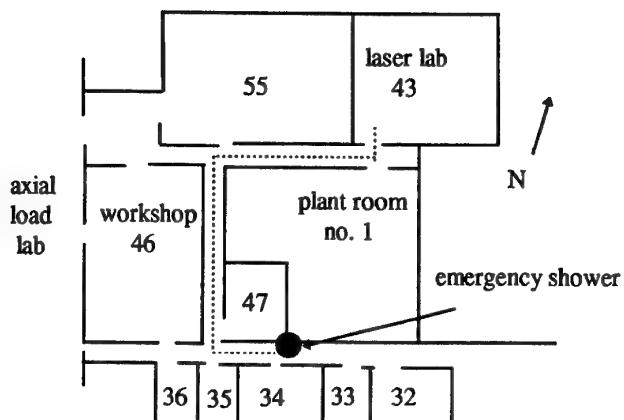
We also participated in a study program (completed December 6, 1996) with the Department of Occupational Health, Safety and Environment at Monash University on the assessment of tritium exposure status in a research facility. As a part of this study regular swabs of the laboratory and the equipment were taken by the researcher as well as the analysis of urine samples which were taken at regular intervals (ie., once per week) and analysed for radiation contamination. If necessary, blood samples were also taken and analysed.

Laboratory coats, safety goggles and double rubber surgical gloves were worn, at all times when working with the tritium.

The Monash researchers informed us that there were some tritium monitors which were available, but they would not have sufficient sensitivity for our purpose, and that their proposed swab tests were the most suitable.

### **Emergency Procedures**

In the case where tritiated water comes in contact with skin, the area should be immediately flushed with large volumes of water in the adjoining sink and the area swabbed before dried. These swabs would then be sent to Monash University as part of the analysis



*Figure A2: Building plan showing position of emergency shower.*

for the study mentioned previously.

In the case where tritiated water comes in contact with eyes or face, the area should be flushed using an eyewash.

In the case where tritiated water comes in contact with clothes, the garment should be immediately removed and sent for analysis.

In the case where a large area of skin or clothing comes into contact with the tritiated water, the nearest emergency shower is located 40 metres away as shown in Figure A2.

If, during the dilution process (see Section A.2), there was a spill of the 1 Ci solution, the clean up of the solution would have included the use of kitty litter to absorb the tritiated water. The kitty litter would then be collected, using a dustpan and brush and placed into a 25 litre plastic bin with a lid and stored for collection by a licensed disposal company.

#### Other Safety Precautions

The handling of any solution of tritiated water was performed in the presence of at least 2 operators.

Staff were not allowed to eat, drink or smoke in the laboratory. Signs indicating this were placed in and around the laboratory, and ingestion and inhalation limits for tritium are listed in Appendix B.

Warning signs were displayed in the laboratory and on entry to the laboratory.

Other users of the laboratory and any other future worker, were made aware of the nature of this work and a copy of this safety study was made available.

The safety and emergency procedures were run through prior to each experiment, and a number of mock runs of the initial dilution process were performed using ordinary water before the commencement of the experiments to build up operator experience and to ensure smooth running of the actual test.

## A.2 Specific Experimental Procedures

### Use of $^3\text{H}$

1. 0.2 ml of tritiated water is received from the supplier in an ampoule in a shielded container, with an activity of 1 Ci. This is stored in a locked refrigerator until required.
2. The 0.2 ml ampoule is opened and the ampoule plus contents are placed into a 1 litre volumetric flask. The flask is then filled with 999.8 ml of distilled water (ie. the 1 litre solution is made up to the mark with distilled water). This solution will be made up inside a fume cupboard, behind a perspex shield. This solution will then be transferred to two 500 ml glass jars and a sample taken by the Monash researcher to check that the activity is 0.5 Ci. To contain any residual tritiated water within the opened ampoule, it will be placed into one of these jars for the duration of the experiments 1 ml of the solution will be placed into 199 ml of a 0.5 N  $\text{H}_2\text{SO}_4$  solution using a plastic pipette. The charging solution will have an activity of 10-3 Ci.
3. Specimens will be charged in this solution for 1 hour. After this time the solution will be flushed down the sink in accordance to the allowable limit of 10 mCi per day (see Appendix 2). After the specimens have been charged they will be stored in a sealed container at  $-10^\circ\text{C}$  to release any excess hydrogen. Preliminary calculations suggest that the activity of the specimens will be 10-8 times that of the solution (which is 1 mCi). Therefore the activity of the specimens, immediately after charging will be approximately 10-8 mCi
4. All personnel will wear laboratory coats, two sets of disposable gloves and safety glasses. The tritiated water will be manipulated behind perspex shields.
5. The only authorised users will be Ms. K. Tsoi and Mr. D. Rowlands.
6. All laboratory benches will be covered with absorbent backed paper.

## Appendix B: Contamination Limits

### Units of activity

The Becquerel (Bq) is the SI unit of the activity of radioactive material. One Bq is equal to one nuclear disintegration (transformation) per second. Activities are often quoted in the non-SI unit the Curie (Ci).  $1 \text{ Ci} = 3.7 \times 10^{10} \text{ Bq}$ .

### Contamination limits

Maximum permissible levels of non-fixed radioactive contamination

|               | Bq/cm <sup>2</sup> | Ci/cm <sup>2</sup> |
|---------------|--------------------|--------------------|
| Beta emitters | 3.7                | 100 pCi            |

The above levels are permissible when averaged over any area of  $100 \text{ cm}^2$  of any part of the surface.

Reference: Annex C to Chapter 6, OPSMAN 2.

Maximum concentration (above natural background) of tritium in air and water

*Table B1: Concentration limits of tritium in air and water*

| Form of isotope | I                           |                               | II                          |                               |
|-----------------|-----------------------------|-------------------------------|-----------------------------|-------------------------------|
|                 | Column 1                    | Column 2                      | Column 1                    | Column 2                      |
|                 | Air<br>(Ci/m <sup>3</sup> ) | Water<br>(Ci/m <sup>3</sup> ) | Air<br>(Ci/m <sup>3</sup> ) | Water<br>(Ci/m <sup>3</sup> ) |
| Soluble         | $4.86 \times 10^{-6}$       | 0.1                           | $2 \times 10^{-7}$          | $2.97 \times 10^{-3}$         |
| Insoluble       | $4.86 \times 10^{-6}$       | 0.1                           | $2 \times 10^{-7}$          | $2.97 \times 10^{-3}$         |
| Submersion*     | 0.002                       |                               | $4.05 \times 10^{-5}$       |                               |

\* Submersion in a semi-infinite cloud of airborne material

Radiation workers are not to be exposed to airborne radioactive material in a concentration which, if averaged over a period of exposure of 40 hours per week for 52 weeks would exceed that set out in Column 1 of I in Table B1.

Contamination on personnel or clothing is not to exceed  $1 \text{ mCi/m}^2$ . Otherwise, appropriate decontamination is to take place before personnel are permitted to leave the work area. This corresponds to the limit given in OPSMAN 2.

Reference: Health (Radiation Safety) Regulations 1984, Schedule 15 and Regulation 608.

**Ingestion Limits for Tritium**

|                                      |         |                  |
|--------------------------------------|---------|------------------|
| Limit of annual intake by ingestion  | 2.6 mCi | member of public |
| Limit of annual intake by inhalation | 1.2 mCi | member of public |
|                                      | 12 mCi  | radiation worker |

Reference: International Atomic Energy Agency's Safety Series No. 9 - "Basic Safety Standards for Radiation Protection", 1982 Edition.

## Appendix C: Disposal of Radioactive Waste

### Disposal of liquid radioactive waste.

Radioactive material is not to be discharged from any premises in a concentration which exceeds the limits specified in II, Columns 1 and 2 of Table B1.

Radioactive material may be discharged into the sewerage system if

- a) the quantity of any radioactive material released into the system in any seven day period does not exceed 20 times the annual limit of intake by ingestion for that radionuclide. (This would mean 52 mCi per week for tritium, or about 10 mCi per day for a 5 day working week),
- b) the quantity of any radioactive material released into the system in any 24 hour period does not exceed the quantity which, if diluted by the average daily quantity of sewage discharged into the system from those premises, would result in an average concentration equal to the appropriate maximum concentration set out in Column 2 of I (Table B1).

Solid radioactive wastes shall be disposed of only in accordance with procedures approved by the Commission. (Regulation 1305). (See below)

Reference: Health (Radiation Safety) Regulations 1984, Regulations 1303-1305.

### Disposal of solid radioactive waste

Treatment as Ordinary Waste.

Provided the activity limits quoted below are not exceeded, solid, liquid and gaseous radioactive waste may be disposed of as ordinary waste by the following means:

- (1) Solid materials may be disposed of in ordinary waste bins provided that the total activity per bin per clearance is less than 1mCi for  $^3\text{H}$  (tritium).

Waste disposed of in this way is to be contained to prevent dispersion before final disposal and is to be inconspicuous compared with other waste in order to minimise the risk of scavenging.

- (2) Sealed tritium gaseous sources may be disposed of in ordinary waste if they are first broken exterior to the building in the open air to allow the gas to escape, provided that the total tritium activity concerned is less than 5 Ci/year.

- (3) Radioactive tritium waste may be deposited at a municipal refuse tip provided each item contains less than 20 mCi.

Reference: Chapter 10: Instructions for the disposal of radioactive materials, OPS-MAN2.

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| 19. ABSTRACT<br>This report introduces a technique known as tritium autoradiography which may be useful in the detection of fatigue damage in metallic components. Tritium autoradiography involves the infusion of radioactive hydrogen (tritium) into a fatigue damaged structure. The damaged areas of the structure may act as preferred trapping sites for the tritium and as the tritium decays via the emission of beta particles it can be detected using nuclear emulsion film. |                              |  |                                    |                     |

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